EXHIBIT E

QUALITY ASSURANCE/QUALITY CONTROL PROCEDURES AND REQUIREMENTS

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1.0 OVERVIEW

1.1 Quality assurance and quality control are integral parts of the Environmental Protection Agency's (EPA) Regional Environmental Analytical Procurement (REAP) program. The quality assurance (QA) process consists of management review and oversight at the planning, implementation, and completion stages of the environmental data collection activity, and ensures that data provided are of the quality required. The quality control (QC) process includes those activities required during data collection to produce the data quality desired and to document the quality of the collected data.

1.2 During the planning of an environmental data collection program, QA activities focus on defining data quality criteria and designing a QC system to measure the quality of data being generated. During the implementation of the data collection effort, QA activities ensure that the QC system is functioning effectively, and that the deficiencies uncovered by the QC system are corrected. After environmental data are collected, QA activities focus on assessing the quality of data obtained to determine its suitability to support enforcement or remedial decisions.

1.3 This exhibit describes the overall QA/QC operations and the processes by which the REAP program meets the objectives defined above. This contract requires a variety of QA/QC activities. These contract requirements are the minimum QC operations necessary to satisfy the analytical requirements associated with the determination of the different method analytes. These QC operations are designed to facilitate laboratory comparison by providing EPA with comparable data from all Contractors. These requirements do not release the analytical Contractor from maintaining their own QC checks on method and instrument performance.

2.0 QA/QC PRACTICES

2.1 Introduction

2.1.1 Appropriate use of data generated under the large range of analytical conditions encountered in environmental analyses requires reliance on the QC procedures and criteria incorporated into the methods. Several of the methods in this contract have been validated on samples typical of those received by the laboratories in the Contract Laboratory Program (CLP). However, the validation of these methods does not guarantee that they perform equally well for all sample matrices encountered. Inaccuracies can also result from causes other than unanticipated matrix effects, such as sampling artifacts, equipment malfunctions, and operator error. Therefore, the quality control component of each method is indispensable.

2.1.2 The data acquired from QC procedures are used to estimate and evaluate the information content of analytical results and to determine the necessity for or the effect of corrective action procedures. The parameters used to estimate information content include precision, accuracy, detection limit, and other quantitative and qualitative indicators. In addition, QC procedures give an overview of the activities required in an integrated program to generate data of known and documented quality required to meet defined objectives.

2.1.3 The necessary components of a complete QA/QC program include internal QC criteria that demonstrate acceptable levels of performance, as determined by QA review. External review of data and procedures is accomplished by the monitoring activities of the EPA. Each external review accomplishes a different purpose. These reviews are described in specific sections of this Exhibit. Laboratory evaluation samples and data packages provide an external QA reference for the program. An on-site evaluation is also part of the external QA monitoring system. A feedback loop provides the results of the various review functions to the Contractors through direct communications with the EPA.

2.2 General QA/QC Practices

2.2.1 The Contractor shall establish a quality assurance program with the objective of providing sound analytical chemical measurements. This program shall incorporate the quality control procedures, any necessary corrective actions, and all documentation required during data collection as well as the quality assessment measures performed by management to ensure acceptable data production.

2.2.2 The primary function of the QA/QC program is the definition of procedures for the evaluation and documentation of sampling and analytical methodologies and the reduction and reporting of data. The objective is to provide a uniform basis for sample collection and handling, instrument and methods maintenance, performance evaluation, and analytical data gathering and reporting. Although it is impossible to address all analytical situations in one document, the approach taken here is to define minimum requirements for all major steps relevant to any analysis performed. In many instances where methodologies are available, specific quality control procedures are incorporated into the method documentation (Exhibit D). Ideally, samples involved in enforcement actions are analyzed only after the methods have met the minimum performance and documentation requirements described in this document.

2.2.3 This exhibit does not provide specific instructions for constructing QA plans, QC systems, or a QA organization. It is, however, an explanation of the QA/QC requirements of the program. It outlines some minimum standards for QA/QC programs. It also includes specific items that are required in a QA plan and by the QA/QC documentation detailed in this contract. Delivery of this documentation provides the Agency with a complete data package which will stand alone, and limits the need for contact with the Contractor or with an analyst, at a later date, if some aspect of the analysis is questioned.

2.2.4 In order to assure that the product delivered by the Contractor meets the requirements of the contract, and to improve interlaboratory data comparison, the Agency requires the following from the Contractor:

- Preparation of and adherence to a written laboratory quality assurance plan, the elements of which are designated in Section 3,
- Preparation of and adherence to QA/QC standard operating procedures as described in Section 4,
- Adherence to the analytical methods and associated QC requirements specified in the contract,
- Verification of analytical standards and documentation of the purity of neat materials and the purity and accuracy of solutions obtained from private chemical supply houses,
- Submission of all raw data and pertinent documentation for Regional review,
- Participation in the analysis of laboratory evaluation samples, including adherence to corrective action procedures,
- Submission, upon request, of a copy of the sample data package,
- Participation in on-site laboratory evaluations, including adherence to corrective action procedures, and
- Submission of all original documentation generated during sample analyses for Agency review.

2.3 SPECIFIC QA/QC PROCEDURES

2.3.1 The quality assurance/quality control (QA/QC) procedures defined herein shall be used by the Contractor when performing the methods specified in Exhibit D. When additional QA/QC procedures are specified in the methods in Exhibit D, the Contractor shall also follow these procedures.

2.3.2 The purpose of this document is to provide a uniform set of procedures for the analysis of inorganic constituents of samples, documentation of methods and their performance, and verification of the sample data generated. The program will also assist laboratory personnel in recalling and defending their actions under cross examination if required to present court testimony in enforcement case litigation.

2.3.3 In this Exhibit, as well as other places within this Statement of Work, the term "analytical sample" is used in discussing the required frequency or placement of certain QA/QC measurements. The term "analytical sample" is defined in the glossary, Exhibit G. As the term is used, analytical sample includes all field samples, including Performance Evaluation Samples received from an external source, but it also includes all required QA/QC samples (matrix spikes, analytical/post-digestion spikes, duplicates, serial dilutions, Laboratory Control Samples (LCS), Interference Check Samples (ICS), Laboratory Fortified Blanks (LFB), preparation blanks and linear range analyses) except those directly related to instrument calibration or calibration verification (calibration standards, ICV/ICB, CCV/CCB). A "frequency of 10%" means once every 10 analytical samples. Note: Calibration verification samples (ICV/CCV) and calibration verification blanks (ICB/CCB) are not counted as analytical samples when determining 10% frequency.

2.3.4 In order for the QA/QC information to reflect the status of the samples analyzed, all samples and their QA/QC analysis shall be analyzed under the same operating and procedural conditions.

2.3.5 If any QC measurement fails to meet contract criteria, the analytical measurement may not be repeated prior to taking the appropriate corrective action as specified in Exhibits E and D.

2.3.6 The Contractor shall report all QC data in the exact format specified in Exhibits B.

2.3.7 Sensitivity, method detection limits (MDLs), precision, linear dynamic range and interference effects shall be established for each analyte on a particular instrument. All reported measurements shall be within the instrumental linear ranges. The analyst shall maintain quality control data confirming instrument performance and analytical results.

2.3.8 Standard laboratory practices for laboratory cleanliness as applied to glassware and apparatus shall be adhered to. Laboratory practices with regard to reagents, solvents, and gases shall also be adhered to. For additional guidelines regarding these general laboratory procedures, see Sections 4 and 5 of the Handbook for Analytical Quality Control in Water and Wastewater Laboratories EPA-600/4-79-019, U.S. EPA Environmental Monitoring Systems Laboratory, Cincinnati, Ohio, March 1979.

3.0 LABORATORY QUALITY ASSURANCE PLAN

3.1 Introduction

The LQAP shall present, in specific terms, the policies, organization, objectives, functional guidelines, and specific QA and QC activities designed to achieve the data quality requirements in this contract. Where applicable, SOPs pertaining to each element shall be included or referenced as part of the LQAP. The LQAP shall be paginated consecutively in ascending order. Additional information relevant to the preparation of a LQAP can be found in Agency and American Society for Testing and Materials publications.

3.2 Required Elements of a Laboratory Quality Assurance Plan

As evidence of such a program, the Contractor shall prepare a written laboratory quality assurance plan (LQAP) which describes the procedures that are implemented to achieve the following:

- Maintain data integrity, validity, and usability,
- Ensure that analytical measurement systems are maintained in an acceptable state of stability and reproducibility,
- Detect problems through data assessment and establish corrective action procedures which keep the analytical process reliable, and
- Document all aspects of the measurement process in order to provide data which are technically sound and legally defensible.

The LQAP shall be available during on-site laboratory evaluation and shall be submitted within 7 days of written request by the EPA. The elements of the LQAP are listed in the following outline.

A. Organization and Personnel

1. QA Policy and Objectives
2. QA Management
    a. Organization
    b. Assignment of QC and QA Responsibilities
    c. Reporting Relationships
    d. QA Document Control Procedures
    e. QA Program Assessment Procedures
3. Personnel
    a. Resumes
    b. Education and Experience Pertinent to this Contract
    c. Training Progress

B. Facilities and Equipment

1. Floor Plan of Facility and Location of Pertinent Instruments
2. Instrumentation and Backup Alternatives
3. Maintenance Activities and Schedules

C. Document Control

1. Laboratory Notebook Policy
2. Sample Tracking/Custody Procedures
3. Logbook Maintenance and Archiving Procedures

4. SDG File Organization, Preparation and Review Procedures
5. Procedures for Preparation, Approval, Review, Revision, and Distribution of SOPs
6. Process for Revision of Technical or Documentation Procedures

D. Analytical Methodology

1. Instrument Calibration Procedures and Frequency
2. Sample Preparation Procedures
3. Sample Analysis Procedures
4. Standards Preparation Procedures
5. Decision Processes, Procedures, and Responsibility for Initiation of Corrective Action
6. Procedures for Monitoring Effectiveness of Corrective Action

E. Data Generation

1. Data Collection Procedures
2. Data Reduction Procedures
3. Data Verification and Completeness Check Procedures
4. Data Reporting and Authorization Procedures

F. Quality Assurance

1. Data Quality Assurance
2. Systems/Internal Audits
3. Performance/External Audits
4. Corrective Action Procedures
5. Monitoring the Effectiveness of Corrective Action
6. Quality Assurance Reporting Procedures
7. Responsibility Designation

G. Quality Control

1. Reagent Check Analysis
2. Reference Material Analysis
3. Internal Quality Control Checks
4. Corrective Action and Determination of QC Limit Procedures
5. Responsibility Designation

H. Safety Programs - Training and Documentation

I. Compliance with Environmental Regulations

1. Air Pollution Prevention Measures
2. Aqueous Effluent Discharge
3. Hazardous and Nonhazardous Waste Management Practices
4. Hazardous Waste Manifesting

3.3 Updating and Submitting the LQAP

3.3.1 Initial Submission:

During the contract solicitation process, the Contractor is required to submit their LQAP to the EPA. Within twenty eight (28) days after contract award, the Contractor shall submit their LQAP which is in compliance with the requirements of this contract to EPA. Within 42 days of receipt of the LQAP, EPA will either provide written comments to the Contractor or approve the LQAP. Within 14 days of receipt of EPA written comments, the Contractor shall submit a revised LQAP which is in compliance with the requirements of this contract. The Contractor shall maintain on file a revised LQAP, fully compliant with the requirements of this contract. The revised LQAP will become the official LQAP under the contract and may be used during legal proceedings. The Contractor shall maintain the LQAP on file at the Contractor's facility for the term of the contract. Both the initial submission and the revised LQAP shall be paginated consecutively in ascending order. The revised LQAP shall include:

3.3.1.1 Changes resulting from A) the Contractor's internal review of their organization, personnel, facility, equipment, policy and procedures and B) the Contractor's implementation of the requirements of the contract; and

3.3.1.2 Changes resulting from the Agency's review of the laboratory evaluation sample data, bidder supplied documentation, and recommendations made during the preaward on-site laboratory evaluation.

3.3.2 Subsequent Updates and Submissions:

During the term of contract, the Contractor shall amend the LQAP when the following circumstances occur:

- The Agency modifies the contract,
- The Agency notifies the Contractor of deficiencies in the LQAP document,
- The Agency notifies the Contractor of deficiencies resulting from the Agency's review of the Contractor's performance,
- The Contractor identifies deficiencies resulting from their internal review of their LQAP document,
- The Contractor's organization, personnel, facility, equipment, policy or procedures change, or
- The Contractor identifies deficiencies resulting from the internal review of their organization, personnel, facility, equipment, policy or procedures changes.

3.3.2.1 The Contractor shall amend the LQAP within 28 days of when the circumstances listed above result in a discrepancy between what was previously described in the LQAP and what is presently occurring at the Contractor's facility.

3.3.2.2 When the LQAP is amended, all changes in the LQAP shall be clearly marked (e.g., a bar in the margin indicating where the change is found in the document, or highlighting the change by underlining the change, bold printing the change, or using a different print font). The amended section pages shall have the date on which the changes were implemented. The Contractor shall incorporate all amendments to the current LQAP document. The Contractor shall archive all amendments to the LQAP document for future reference by the Agency. The Contractor shall send a copy of the current LQAP document within 7 days of a written request by the EPA as directed.

3.4 Corrective Actions

If a Contractor fails to adhere to the requirements listed in this section, a Contractor may expect, but the Agency is not limited to the following actions: reduction in the numbers of samples sent under this contract, suspension of sample shipment to the Contractor, data package

audit, an on-site laboratory evaluation, remedial performance evaluation sample, and/or contract sanctions, such as a Cure Notice.

4.0 STANDARD OPERATING PROCEDURES

4.1 Introduction

In order to obtain reliable results, adherence to prescribed analytical methodology is imperative. In any operation that is performed on a repetitive basis, reproducibility is best accomplished through the use of Standard Operating Procedures (SOPs). As defined by the EPA, an SOP is a written document which provides directions for the step-by-step execution of an operation, analysis, or action which is commonly accepted as the method for performing certain routine or repetitive tasks.

SOPs prepared by the Contractor shall be functional (i.e., clear, comprehensive, up-to-date, and sufficiently detailed to permit duplication of results by qualified analysts). The SOPs shall be paginated consecutively in ascending order.

All SOPs shall reflect activities as they are currently performed in the laboratory. In addition, all SOPs shall be:

- Consistent with current EPA regulations, guidelines, and the REAP contract's requirements.
- Consistent with instruments manufacturers' specific instruction manuals.
- Available to the EPA during an on-site laboratory evaluation. A complete set of SOPs shall be bound together and available for inspection at such evaluations. During on-site laboratory evaluations, laboratory personnel may be asked to demonstrate the application of the SOPs.
- Available to the EPA within 7 days of a written request.
- Capable of providing for the development of documentation that is sufficiently complete to record the performance of all tasks required by the protocol.
- Capable of demonstrating the validity of data reported by the Contractor and explain the cause of missing or inconsistent results.
- Capable of describing the corrective measures and feedback mechanism utilized when analytical results do not meet protocol requirements.
- Reviewed regularly and updated as necessary when contract, facility, or Contractor procedural modifications are made.
- Archived for future reference in usability or evidentiary situations.
- Available at specific work stations as appropriate.
- Subject to a document control procedure which precludes the use of outdated or inappropriate SOPs.

4.2 SOP Format:

The format for SOPs may vary depending upon the kind of activity for which they are prepared, however, at a minimum, the following sections shall be included:

- Title Page
- Scope and Application
- Definitions
- Procedures
- Quality Control (QC) Limits
- Corrective Action Procedures, Including Procedures for Secondary Review of Information Being Generated
- Documentation Description and Example Forms
- Miscellaneous Notes and Precautions
- References

4.3 SOP Requirements

The Contractor shall maintain the following SOPs:

1. Evidentiary SOP

   Evidentiary SOPs for required chain-of-custody and document control are discussed in Exhibit F.

   a. Sample receiving and documentation procedures
   b. Sample identification and documentation procedures
   c. Sample security and security precautions
   d. Sample storage and documentation procedures
   e. Sample tracking and document control
   f. Computer resident sample data control
   g. Refrigerator temperature logbooks
   h. Sample digestate/distillate storage logbooks

2. Sample Preparation
3. Glassware Cleaning
4. Calibration (Balances, etc.)
   a. Procedures for verifying calibration
   b. Frequency requirements
   c. Preventative maintenance schedule and procedures
   d. Acceptance criteria and corrective actions
   e. Logbook maintenance authorization
5. Analytical Procedures (for each analytical system)
   a. Instrument performance specifications
   b. Instrument operating procedures
   c. Data acquisition system operation

d. Procedures when automatic quantitation algorithms are overridden

e. QC required parameters

f. Analytical run/injection logbooks

g. Instrument error and editing flag descriptions and resulting corrective actions

6. Maintenance activities (for each analytical system)

a. Preventative maintenance schedule and procedures

b. Corrective maintenance determinants and procedures

c. Maintenance authorization

7. Analytical standards

a. Standard coding/identification and inventory system

b. Standards preparation logbook(s)

c. Standard preparation procedures

d. Procedures for equivalency/traceability analyses and documentation

e. Purity logbook (primary standards and solvents)

f. Storage, replacement, and labelling requirements

g. QC and corrective action measures

8. Data reduction procedures

a. Data processing systems operation

b. Outlier identification methods

c. Identification of data requiring corrective action

d. Procedures for format and/or forms for each operation

9. Documentation policy/procedures

a. Laboratory/analyst's notebook policy, including review policy

b. Complete SDG File contents

c. Complete SDG File organization and assembly procedures, including review policy

d. Document inventory procedures, including review policy

10. Data verification/self-inspection procedures

    a. Data flow and chain-of-command for data review

    b. Procedures for measuring precision and accuracy

    c. Evaluation parameters for identifying systematic errors

    d. Procedures to assure that hardcopy deliverables are complete and compliant with the requirements in SOW Exhibits B.

    e. Demonstration of internal QA inspection procedure (demonstrated by supervisory and QA sign-off on personal notebooks, internal laboratory evaluation samples, data package (CSF) deliverables, etc.).

    f. Frequency and type of internal audits (e.g., random, quarterly, spot checks, perceived trouble areas).

    g. Demonstration of problem identification, corrective actions and resumption of analytical processing. Sequence resulting from internal audit (i.e., QA feedback).

    h. Documentation of audit reports (internal and external), response, corrective action, etc.

11. Data management and handling

    a. Procedures for controlling and estimating data entry errors.

    b. Procedures for reviewing changes to data and deliverables and ensuring traceability of updates.

    c. Lifecycle management procedures for testing, modifying and implementing changes to existing computing systems including hardware, software, and documentation or installing new systems.

    d. Database security, backup and archival procedures including recovery from system failures.

    e. System maintenance procedures and response time.

    f. Individuals(s) responsible for system operation, maintenance, data integrity and security.

    g. Specifications for staff training procedures.

4.4 Updating and Submitting the SOPs:

4.4.1 Initial Submission:

During the contract solicitation process, the Contractor is required to submit their SOPs to the EPA. Within twenty eight (28) days after contract award, the Contractor shall submit a complete set of SOPs which is in compliance with the requirements of this contract to EPA. Within 42 days of receipt of the SOPs, EPA will either provide written comments to the Contractor or approve the SOPs. Within 14 days of receipt of EPA written comments, the Contractor shall submit a complete revised set of SOPs which are in compliance with the requirements of this contract. The Contractor shall maintain on file a complete revised set of SOPs, fully compliant with the requirements of this contract. The revised SOPs will become the official SOPs under the contract and may be used during legal proceedings. The Contractor shall maintain the complete set of SOPs on file at the Contractor's facility for the term of the contract. Both the initial submission of SOPs and the revised SOPs shall be paginated consecutively in ascending order. The revised SOPs shall include:

4.4.1.1 Changes resulting from A) the Contractor's internal review of their procedures and B) the Contractor's implementation of the requirements of the contract; and

4.4.1.2 Changes resulting from the Agency's review of the laboratory evaluation sample data, bidder supplied documentation, and

recommendations made during the preaward on-site laboratory evaluation.

4.4.2 Subsequent Updates and Submissions:

During the term of contract, the Contractor shall amend the SOPs when the following circumstances occur:

- The Agency modifies the contract,
- The Agency notifies the Contractor of deficiencies in their SOPs documentation,
- The Agency notifies the Contractor of deficiencies resulting from the Agency's review of the Contractor's performance,
- The Contractor's procedures change,
- The Contractor identifies deficiencies resulting from the internal review of their SOPs documentation, or
- The Contractor identifies deficiencies resulting from the internal review of their procedures.

4.4.2.1 Existing SOPs shall be amended or new SOPs shall be written within 28 days of when the circumstances listed above result in a discrepancy between what was previously described in the SOPs and what is presently occurring at the Contractor's facility. All changes in the SOPs shall be clearly marked (e.g., a bar in the margin indicating where the change is in the document, or highlighting the change by underlining the change, bold printing the change, or using a different print font). The amended/new SOPs shall have the date on which the changes were implemented.

4.4.2.2 When existing SOPs are amended or new SOPs are written, the Contractor shall document the reasons for the changes, and maintain the amended SOPs or new SOPs on file. Documentation of the reasons for the changes shall be maintained on file with the amended SOPs or new SOPs.

4.4.2.3 The Contractor shall send a complete set of current SOPs within 7 days of a written request by the EPA as directed.

4.4.2.4 Documentation of the reasons for changes to the SOPs shall also be submitted along with the SOPs. An alternate delivery schedule for submitting the letter and amended/new SOPs may be proposed by the Contractor, but it is the sole decision of the Agency to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the EPA why he/she is unable to meet the delivery schedule listed in this section. The EPA will not grant an extension for greater than 28 days for amending/writing new SOPs. The EPA will not grant an extension for greater than 14 days for submission of the letter documenting the reasons for the changes and for submitting amended/new SOPs. The Contractor shall proceed and not assume that an extension will be granted until so notified by the EPA.

4.5 Corrective Actions

If a Contractor fails to adhere to the requirements listed in this section, a Contractor may expect, but the Agency is not limited to the following actions: reduction in the number of samples sent under this contract, suspension of sample shipment to the Contractor, data package audit, on-site laboratory evaluation, remedial performance evaluation sample, and/or contract sanctions, such as a Cure Notice.

5.0 REQUIRED QA/QC OPERATIONS

This section outlines the minimum QA/QC operations necessary to satisfy the analytical requirements of the contract for the target analyte metals, cyanide, total organic carbon, total combustible organics, and grain size distribution. The following QA/QC operations shall be performed as described in this Exhibit:

5.1 Sample Analysis

5.2 Instrument Calibration

5.3 Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) - Form 2A

5.4 Laboratory Forified Blank (LFB) - Form 2B

5.5 Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB) and Preparation Blank (PB) - Form 3

5.6 ICP Interference Check Sample (ICS) Analyses - Form 4

5.7 Spike Sample Analysis (S) - Form 5A, and Post-digestion/post-distillation Spike Analysis - Form 5B

5.8 Duplicate Sample Analysis (D) - Form 6

5.9 Laboratory Control Sample (LCS) Analysis - Form 7

5.10 ICP Serial Dilution Analysis (L) - Form 9

5.11 SDG-Specific Performance Evaluation Samples (PES)

5.12 Method Detection Limit (MDL) Determination - Forms 10A/B/C

5.13 Interelement Corrections for ICP (IEC) - Forms 11A/B

5.14 Linear Range Analysis (LRA) - Form 12

5.15 Furnace AA QC Analyses - Forms 8, 14

5.16 Tables/Diagrams

All references to the CRQLs refer to the CRQLs, specified in Exhibit C, which are associated with the concentration level (Medium or Low) and/or matrix of the sample as designated on the Traffic Report. In addition, all references to the Low Level CRQL apply only to Low Level aqueous samples.

For the QA/QC operations necessary to satisfy the analytical requirements of the contract for TOC, TCO, and grain size distribution, refer to the specific methods in Exhibit D in addition to the following requirements.

5.1 Sample Analysis

5.1.1 After sample preparation, all final prepared digestates/distillates ready for analysis shall be analyzed initially without any dilution. If the analysis yields a result which exceeds the linear range of the ICP, or the calibrated range of the other instruments, the Contractor shall use the least dilution necessary to bring the analyte concentration within the linear or calibrated range of the instrument, as appropriate, but not below the CRQL.

5.1.2 Whenever the ICP is utilized to analyze and report one or more target analytes, then aluminum, calcium, iron, magnesium, and all other analytes which were determined to be a spectral interference (as specified in Exhibit E, section 5.13) shall also be analyzed and reported with the samples in the SDG in accordance with the requirements of this contract.

5.1.3 Report sample concentrations on Form I as specified in Exhibit B. If a sample required dilution and/or preconcentration for a target analyte, the sample concentration must be corrected for all dilution

and preconcentration factors, which were applied to the sample and analyte, before reporting the result on Form I. The preconcentration factors and dilution factors shall be reported on Forms I and XIV as described in Exhibit B.

5.2 Instrument Calibration

5.2.1 Guidelines for instrumental calibration are given in Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, and/or Exhibit D. Calibrate all instruments according to the instrument manufacturer's instructions or as specified in Exhibit D. Instruments shall be calibrated daily or once every 24 hours and each time the instrument is set up. The date and time of instrument calibration must be reported in the raw data. The number of calibration standards required for each analytical system are as follows:

5.2.1.1 ICP systems must be calibrated using at least one blank and one standard that is within the established linear range for the target analyte.

5.2.1.2 Flame AA and furnace AA systems must be calibrated using at least one blank and three standards. One calibration standard must be at a concentration equal to the CRQL and the remaining two standards must be in graduated amounts that define the linear range for the target analyte as defined in the individual methods in Exhibit D.

5.2.1.3 Mercury and cyanide systems must be calibrated using at least one blank and five standards. One calibration standard must be at a concentration equal to the CRQL and the remaining four standards must be in graduated amounts that define the linear range for the target analyte.

5.2.1.4 Total Organic Carbon analyzers must be calibrated using at least one blank and four standards. One calibration standard must be at a concentration equal to the CRQL and the remaining four standards must be in graduated amounts that define the linear range of the instrument.

5.2.2 Calibration standards must be prepared by diluting stock solutions at the time of analysis. (Note: Mercury standards are prepared at the time of sample preparation). Calibration standards must be discarded after use. For ICP analysis, follow the calibration procedures in Exhibit D. The date and time of the preparation and analysis of the standards must be reported in the raw data.

5.2.3 If multiple sets of analytical conditions are used to measure and report results for an analyte, the instrument must also be calibrated and reported for each set of analytical conditions.

5.2.4 Baseline correction is acceptable provided it is performed for every standard, sample, QC sample, and required blank analyzed.

5.2.5 Calibration curves with three or more points must use a linear least squares fit and must have a correlation coefficient greater than or equal to 0.995 before any further analysis may continue. No other calibration curves (second order, third order, etc.,) shall be acceptable.

5.2.5.1 If the calibration curve yields a correlation coefficient less than 0.995, terminate the analysis, correct the problem, recalibrate the instrument, and reverify the correlation coefficient. The correlation coefficient for each calibration curve must be clearly documented in the raw data and must be submitted with the data package.

5.2.6 Sample results reported with a non-compliant instrument calibration shall receive a commensurate reduction in sample price or zero payment due to data rejection depending upon the impact of the non-compliance on data usability.

5.3 Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)

5.3.1 Initial Calibration Verification (ICV)

5.3.1.1 Immediately after calibration and prior to the analysis of any samples, QC samples or required blanks, the accuracy of the initial calibration must be verified and documented by the analysis of a separate source Initial Calibration Verification standard(s). An ICV analysis shall be analyzed for every target analyte reported. The analysis conditions for the ICV must be the same as those for the analytical samples. If multiple sets of analytical conditions are used to measure and report results for an analyte, the ICV must also be measured and reported for each set of analytical conditions.

5.3.1.2 ICV standards can be obtained from commercial vendors, or prepared in-house. The source of the ICV solution for each target analyte must be independent of the source used for the instrument calibration standards. The concentration of a target analyte in the ICV shall be within the low to mid range of the calibration and at a concentration other than that used for instrument calibration. The Contractor shall maintain documentation of the purity confirmation of the material to verify the integrity of the standard solutions.

5.3.1.3 For all instrumental techniques, the Initial Calibration Verification Solution(s) shall be run at each wavelength used for analysis.

5.3.1.4 For aqueous cyanide samples, the ICV solution may also serve as the LCS and must be distilled prior to analysis. Furthermore, it must be distilled with the batch of samples to be analyzed (i.e., for which it serves as the LCS). For soil cyanide samples, a separate LCS shall be required in addition to the ICV standard.

5.3.1.5 For aqueous TOC samples, the ICV solution may also serve as the LCS. For soil TOC samples, a separate LCS shall be required in addition to the ICV standard.

5.3.1.6 Calculate the percent recovery for each analyte in the ICV analysis using Equation 1. If the measurements exceed the control limits of Table 1 (in Exhibit E), Initial and Continuing Calibration Verification Control Limits for Inorganic Analyses, the analysis shall be terminated, the problem corrected, the instrument recalibrated, and the new calibration verified.

EQ. 1

$$\textit{Percent Recovery}\ (\%R) = \frac{M}{T}\ x\ 100$$

Where,

M = Measured analyte concentration

5.5.1.5 Sample results reported with a non-compliant initial calibration blank shall receive a commensurate reduction in sample price or zero payment due to data rejection depending upon the impact of the non-compliance on data usability.

5.5.2 Continuing Calibration Blank (CCB)

5.5.2.1 The continuing calibration blank shall be analyzed (at each wavelength used for analysis) immediately after every continuing calibration verification. The CCB is used to monitor the accuracy of the calibration at a concentration of "0" throughout the analytical run sequence, and check for carryover from the previous analytical samples. The analysis conditions for the CCB must be the same as those for the analytical samples. Unless otherwise specified in Exhibit D, the continuing calibration blank is in the same matrix as the initial calibration standards. If multiple sets of analytical conditions are used to measure and report results for a target analyte, the CCB must also be measured and reported for each set of analytical conditions.

5.5.2.2 Calculate the concentration of each target analyte of concern in the CCB analysis. If the absolute value of the continuing calibration blank result is greater than or equal to the MDL, the result shall be reported as specified in Exhibit B. If the absolute value of the calibration blank result is greater than or equal to the CRQL (Exhibit C) or 3xMDL, whichever is lower, terminate the analysis, correct the problem, recalibrate the instrument, reanalyze the ICV, ICB, LFB, ICS (ICP only), CCV, CCB, and reanalyze all of the analytical samples analyzed since the last compliant CCB for the affected analytes.

5.5.2.3 The results for the continuing calibration blank shall be recorded on FORM III as indicated in Exhibit B.

5.5.2.4 Continuing calibration blank criteria must be met before any samples, QC samples, or required blanks are analyzed. Any samples, QC samples or required blanks analyzed when the continuing calibration blank criteria have not been met shall require reanalysis at no additional cost to the Agency. Reanalyses must be performed within contract required holding times and must meet all sample acceptance criteria.

5.5.2.5 Sample results reported with a non-compliant continuing calibration blank shall receive a commensurate reduction in sample price or zero payment due to data rejection depending upon the impact of the non-compliance on data usability.

5.5.3 Preparation Blank (PB) Analysis

5.5.3.1 At least one preparation blank shall be prepared and analyzed with every Sample Delivery Group (SDG) or with each batch[1] of samples prepared, whichever is more frequent. The preparation blank, consisting of distilled or deionized water for aqueous samples and appropriate blank matrix (blank soil or blank sand) for soil/sediment/solid samples, shall be processed through the entire sample preparation and analysis procedures (refer to the individual methods in Exhibit D). If more than one matrix exists within an SDG, a separate preparation blank per matrix must be prepared. Preparation blanks must be analyzed in the same analytical run sequence as the samples with which the blanks were prepared.

5.5.3.2 Determine the preparation blank concentration of each analyte to be reported.

5.5.3.2.1 If the absolute value of the concentration of each target analyte in the blank is less than the Contract Required Quantitation Limit (Exhibit C) or 3xMDL, whichever is lower, no corrective action is required.

[1]A group of samples prepared at the same time.

5.5.3.2.2 If any target analyte concentration in the blank is greater than or equal to the CRQL or 3xMDL, whichever is lower, the lowest concentration that can be reported for that analyte in the associated samples must be greater than or equal to 10x the blank concentration. Otherwise, all samples associated with the blank, where the analyte's concentration is less than 10x the blank concentration and greater than or equal to the CRQL or 3xMDL (whichever is lower), must be re-prepared and reanalyzed for that analyte.

5.5.3.2.3 If the concentration of the blank is at or below the negative CRQL or 3xMDL, whichever is lower, then all samples reported at or below 10xCRQL or 10x(3xMDL), whichever is lower, associated with the blank shall be redigested and reanalyzed.

5.5.3.3 Low Level Aqueous Samples

5.5.3.3.1 The preparation blank shall be subject to the same procedures and conditions used for the associated batch of Low Level aqueous samples. The preparation blank must be reduced and adjusted to the same final volumes and, thus, have the same preconcentration factor as the associated samples for each target analyte.

5.5.3.4 The results for the preparation blank shall be recorded in ug/L for aqueous samples and in mg/Kg for solid samples on FORM III.

5.5.3.5 Preparation blank criteria must be met before any samples or QC samples are analyzed. Any samples or QC samples analyzed when the preparation blank criteria have not been met must be redigested and reanalyzed at no additional cost to the Agency. Reanalyses must be performed within contract required holding times and must meet all sample acceptance criteria.

5.5.3.6 Sample results reported with a non-compliant preparation blank shall receive a commensurate reduction in sample price or zero payment due to data rejection depending upon the impact of the non-compliance on data usability.

5.6 ICP Interference Check Sample (ICS) Analysis

Inherent in ICP analyses are interelement correction factors and spectral background corrections. To verify the accuracy of the background correction points and interelement correction factors, the Contractor shall, for one or more target analytes reported from the ICP, analyze and report the results for the ICP Interference Check Samples at the beginning and end of the analytical sequence but not less than twice per 8 hour working shift. The analysis conditions for the ICS must be the same as those for the analytical samples. If multiple sets of analytical conditions are used to measure and report results for an analyte, the ICS must also be measured and reported for each set of analytical conditions.

5.6.1 The Interference Check Samples consist of two solutions: Solution A and Solution AB. Solution A consists of the interferents, and Solution AB consists of the analytes mixed with the interferents. An ICS analysis consists of analyzing both solutions consecutively (starting with Solution A) for all wavelengths used for each target analyte reported by ICP. The ICS analysis shall follow immediately after the LFB analysis and precede the CCV analysis. (Thus for ICP, the analytical run sequence shall be ICV, ICB, LFB, ICS-A, ICS-AB, CCV, CCB, 10 analytical samples, CCV, CCB... last analytical sample, LFB, ICS-A, ICS-AB, CCV, CCB.)

5.6.2 The ICS solutions can be obtained from a commercial vendor, or prepared in-house. If prepared in-house, the ICS solutions must be prepared with ultra high purity grade chemicals and at the concentration levels specified in Table 2 (in Exhibit E), Interferent and Analyte Elemental Concentrations Used for ICP Interference Check Sample. The ICS-A contains the interferents only, and ICS-AB contains both the interferents and the analytes. The Contractor shall maintain documentation of the purity confirmation of the material to verify the integrity of the standard solutions.

5.6.3 Dilution of the ICS-A or ICS-AB solution prior to analysis is permitted only if an analyte or interferent concentration exceeds the linear range of the instrument. When dilution is necessary, the dilution factor must be kept to a minimum. Results from the diluted ICS analysis are reported only for the specific analytes/interferents which require dilution. Results for the other analytes/interferents must be reported from the undiluted ICS analysis. When dilution is required, the ICS analysis run sequence must be undiluted ICS-A, undiluted ICS-AB, diluted ICS-A, and diluted ICS-AB. If the interferent requiring dilution interferes on an analyte which does not require dilution, then the interference correction must be performed using the actual concentration of the interferent (as determined from the diluted sample) and the concentration of the analyte found in the undiluted sample. Interference corrections must be made based on the actual concentration of the interferent and not the apparent concentration obtained when the interferent concentration is above the linear range.

5.6.4 Determine the concentration of every interferent and analyte (listed in Table 2) in the ICS-A solution.

5.6.4.1 Calculate the percent recovery for each interfering analyte using Equation 1. The percent recovery of the interferents in the ICS-A solution must be within the control limits of 80-120%, inclusive. If the percent recovery of the interferents is within the control limits, report the result as specified in Exhibit B.

5.6.4.2 The value of the non-interfering analyte concentrations must be within the control limits of ± 3xMDL or ± CRQL, whichever is less, of their ICS-A true value (the true value shall be zero unless otherwise stated). If the value of the non-interfering analyte is within the control limits, the result shall be reported as specified in Exhibit B.

5.6.5 Determine the concentration of every interferent and analyte (listed on Table 2) for the ICS-AB solution and calculate the percent recovery of each using Equation 1. The percent recovery of the analytes/interferents in the ICS-AB solution must be within the control limits of 80-120%, inclusive, for all analytes.

5.6.6 Corrective action must be performed when any of the following control limits are exceeded:

- the percent recovery of the interferents in the ICS-A solution are outside the 80-120% recovery control limits,
- the results for the non-interfering analytes in the ICS-A solution are not within the control limits of the true value ± 3xMDL or ± CRQL, whichever is less, or
- the percent recovery of the analytes/interferents in the ICS-AB solution is outside the 80-120% recovery control limits.

5.6.7 If the control limits are not met for the analytes or interferents in the ICS-A and/or ICS-AB solutions, terminate the analysis, correct the problem, recalibrate the instrument, and reanalyze all of the analytical samples, within a valid analytical run sequence, that followed the last compliant ICS (ICS-A and ICS-AB) analyses.

5.6.8 Report the measured concentration, the true value, the percent recoveries, and the appropriate analyte control limits for the initial and subsequent ICS analyses on Form IVs, as indicated in Exhibit B.

5.6.9 Interference Check Sample (ICS) criteria must be met before any samples, QC samples, or required blanks are analyzed. Any samples, QC samples or required blanks analyzed when the ICS criteria have not been met shall require reanalysis at no additional cost to the Agency. Reanalyses must be performed within contract required holding times and must meet all sample acceptance criteria.

5.6.10 Sample results reported with a non-compliant ICS analysis shall receive a commensurate reduction in sample price or zero payment due to data rejection depending upon the impact of the non-compliance on data usability.

5.7 Spike Sample Analysis (S) and Post-digestion/post-distillation Spike Analysis

5.7.1 The spike sample analysis is designed to provide information about the effect of the sample matrix on the digestion and/or measurement methodology. At least one spike sample analysis (matrix spike) shall be performed on each group of samples of a similar matrix type (i.e., aqueous, soil) and concentration (i.e., low, medium) or for each Sample Delivery Group.[2] The spike must be added prior to the addition of any reagents and before any digestion, distillation, or sample preparation steps. If two or more methods are used to determine and report a target analyte, then the matrix spike sample must be analyzed by each method.

5.7.1.1 Samples identified as equipment blanks shall not be used for spiked sample analysis.

5.7.1.2 As designated on the sample chain-of-custody, EPA may require that a specific sample be used for the spike sample analysis. Unless otherwise specified, the same field samples shall be used for both the matrix spike and duplicate analyses.

[2] Upon request, EPA may require additional spike sample analyses, for which the Contractor shall be paid.

5.7.1.3 When the spike analysis is performed on the same sample chosen for the duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample" (see Section 5.8, Duplicate Sample Analysis). The average of the duplicate results must not be used for the purpose of determining percent recovery.

5.7.2 The analyte spike shall be added in the amounts specified in Tables 3 and 4 depending on the matrix and concentration level. Note: See Table 3 footnotes for concentration levels and applications.

5.7.2.1 Low Level Aqueous Samples

5.7.2.1.1 The matrix spike sample shall be subject to the same procedures and conditions used for the associated batch of samples for each target analyte. The matrix spike sample must be reduced and adjusted to the same final volumes and, thus, have the same preconcentration factor as the associated samples for each target analyte.

5.7.2.1.2 The analyte spike shall be added in the amount specified in Table 4 - Spiking Levels for Low Level Aqueous Samples.

5.7.3 Determine the concentration of each analyte in the original and matrix spike samples and calculate the percent recovery using Equation 3.

EQ.3

$$\%\ Recovery = \frac{SSR - SR}{SA} \times 100$$

Where,

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

When sample concentration is less than the method detection limit, use SR = 0 only for purposes of calculating % Recovery.

5.7.4 The units for reporting spike sample results shall be identical to those used for reporting sample results on FORM I (i.e., ug/L for aqueous samples or mg/Kg dry weight for soil/sediment/solid samples).

5.7.5 For sample results where the analyte concentration in the original sample is less than or equal to 4x the amount of spike added, the control limits for percent recovery shall be 75-125%, inclusive. When the sample result is greater than 4x the amount of spike added, there are no defined control limits.

5.7.6 The spike sample results (sample results and % recovery results, positive or negative) shall be reported on FORM V, as indicated. If an analyte spike recovery is not at or within the limits of 75-125%, flag that analyte with an "N" on FORM V and also on FORM I for all samples associated with that spike sample and determined by the same analytical method. An exception to this rule is granted in situations where the sample concentration exceeds the spike concentration by more than a factor of four. In such an event, the data shall be reported unflagged even if the percent recovery does not meet the 75-125% recovery criteria.

5.7.7 In the instance where there is more than one spike sample per matrix and concentration per method per SDG, if one spike sample recovery is not within contract criteria, flag all the samples of the same matrix, level, and method in the SDG.

5.7.8 Spike samples must be prepared and analyzed along with the other samples, QC samples, and required blanks in an SDG. If spike samples are not prepared and analyzed with the samples, QC samples or required blanks in an SDG, the contractor shall reprepare and

reanalyze the required number of spike samples, including their original samples and all associated QC samples, at no additional cost to the Agency. Reanalyses must be performed within contract required holding times and must meet all sample acceptance criteria.

5.7.9 Sample results reported without spike analyses shall receive a commensurate reduction in sample price depending upon the impact of the non-compliance on data usability.

5.7.10 Post-digestion/post-distillation Spike Analysis

5.7.10.1 For flame AA, ICP, and CN analyses, when the pre-digestion/pre-distillation spike recovery falls outside the control limits and the sample result does not exceed 4x the spike added, a post-digestion/post-distillation spike shall be performed for those elements that do not meet the specified criteria. Spike the unspiked aliquot of the sample at 2x the indigenous level or 2x CRQL, whichever is greater. Results of the post-digestion/post-distillation spike shall be reported on FORM V (B). Note: No post-digest spike is required for Hg.

5.7.10.2 Post-digestion/post-distillation spike samples must be prepared and analyzed for all spiked elements not meeting the control limit criteria. If required post-digestion/post-distillation spike samples are not prepared and analyzed during the original analytical run sequence, the contractor shall prepare and analyze the post-digestion/post-distillation spike samples along with the original sample and the original spiked sample, during a valid analytical sequence, at no additional cost to the Agency. Reanalyses must be performed within contract required holding times and must meet all sample acceptance criteria.

5.7.10.3 Sample results reported without post-digestion/post-distillation spike sample analyses shall receive a commensurate reduction in sample price depending upon the impact of the non-compliance on data usability.

5.8 Duplicate Sample Analysis (D)

5.8.1 One duplicate sample shall be analyzed for each group of samples of the same matrix (i.e., aqueous or soil/sediment/solid) and concentration (i.e., low, medium) or for each Sample Delivery Group, whichever is greater.[3] The duplicate sample analysis provides information regarding the precision of the preparation and analysis procedures. If the original sample analyte concentrations are determined and reported by more than one method (i.e., ICP, GFAA), the duplicate results for those analytes shall also be determined and reported using those same methods. Duplicates shall not be averaged for reporting on FORM I.

5.8.1.1 Duplicate sample analyses are required for percent solids for sample analyses where percent solids determination is required.

5.8.1.2 Samples identified as equipment blanks must not be used for the duplicate sample analysis.

5.8.1.3 As designated on the sample chain-of-custody, the EPA may require that a specific sample be used for the duplicate sample analysis. Unless otherwise specified, the same field sample shall be used for both the matrix spike and duplicate sample analyses.

5.8.2 Determine the concentration of each analyte in both the original and duplicate samples. Results for duplicate sample analyses shall be reported on FORM VI in ug/L for aqueous samples and mg/Kg dry weight basis for soil/sediment/solid samples.

5.8.3 Calculate the Relative Percent Difference (RPD) between the original and duplicate sample concentrations using Equation 2. If both sample

[3]Upon request, EPA may require additional duplicate sample analyses, for which the Contractor will be paid.

values are less than the MDL, then the RPD is not calculated on FORM VI.

EQ. 2

$$RPD = \frac{|S - D|}{(S+D)/2} \times 100$$

Where,

RPD = Relative Percent Difference
S = Sample Concentration (original)
D = Duplicate Sample Concentration

5.8.4 If an analyte's duplicate result is outside the control limits specified below, flag that analyte with an "*" on FORM VI and also on FORM I for all samples received associated with that duplicate sample. When there is more than one duplicate sample per SDG, and one duplicate result is not within contract criteria, flag all samples of the same matrix, concentration, and method in the SDG.

5.8.4.1 If the analyte concentration in both the original sample and duplicate sample is greater than 5x CRQL, then the calculated RPD shall be used to evaluate the duplicate precision. The RPD result for each analyte must be less than or equal to 20%.

5.8.4.2 If the analyte concentration in one of the samples (original or duplicate) is greater than 5x CRQL and the other is less than or equal to 5x CRQL, then the absolute value of the difference between the two concentrations shall be used to evaluate duplicate precision and the CRQL shall define the control limit. In this case, enter the CRQL in the field "Control Limit" column on FORM VI. (Note, for soil/sediment/solid sample results the CRQL must be adjusted for original sample weight and percent solids.) The absolute value difference for each analyte must be less than or equal to the CRQL.

5.8.4.3 If the analyte concentration in both the original sample and duplicate sample is less than the CRQL, then no control limit is applicable.

5.8.5 Low Level Aqueous Samples

5.8.5.1 The duplicate sample shall be subject to the same procedures and conditions used for the associated batch of samples for each target analyte. The duplicate sample must be reduced and adjusted to the same final volumes and thus, have the same preconcentration factor as the associated Low Level aqueous samples for each target analyte.

5.8.6 Duplicate samples must be prepared and analyzed along with the other samples, QC samples, and required blanks in an SDG. If duplicates are not prepared and analyzed with the samples, QC samples or required blanks in an SDG, the contractor shall reprepare and reanalyze the required number of duplicates, including the originals, at no additional cost to the Agency. Reanalyses must be performed within contract required holding times and must meet all sample acceptance criteria.

5.8.7 Sample results reported without duplicate analyses shall receive a commensurate reduction in sample price depending upon the impact of the non-compliance on data usability.

5.9 Laboratory Control Sample (LCS) Analysis

5.9.1 Aqueous and solid Laboratory Control Samples (LCS) shall be analyzed for each analyte using the same sample preparations, analytical methods and QA/QC procedures employed for the EPA samples received. The LCS sample analysis provides information regarding the accuracy of the preparation and analysis procedures. One LCS sample is prepared and analyzed with each SDG or batch of samples prepared, whichever is greater. If more than one matrix exists within an SDG, a matrix specific LCS must be prepared for each type of matrix. If sample analyte concentrations are determined and reported by more than one method, the LCS results for those analytes shall also be determined and reported using those same methods.

5.9.2 The LCS sample may be obtained by a commercial vendor, or prepared in-house. If obtained commercially, the vendor must certify the analyte concentrations against NIST-traceable standards. If prepared in-house, the concentration of the analyte in the source material must be certified against NIST-traceable standards.

5.9.3 For aqueous LCS solutions, the initial Calibration Verification solution may be used. Note, for cyanide analysis this solution must be distilled. The concentration of the analytes in an aqueous LCS solution shall be within the low to mid range of the calibration for the method of analysis.

5.9.4 Low Level Aqueous Samples

5.9.4.1 The aqueous Laboratory Control Sample shall be subject to the same procedures and conditions used for the associated batch of samples for each target analyte. The LCS must be reduced and adjusted to the same final volumes and, thus, have the same preconcentration factor as the associated Low Level aqueous samples for each target analyte.

5.9.4.2 If prepared in-house, the LCS shall be spiked in the amount provided in Table 4 - Spiking Levels for Low Level Aqueous Samples and Laboratory Control Sample.

5.9.5 The solid LCS shall be prepared and analyzed using each of the procedures applied to the samples received (exception: percent solids determination not required). The solid LCS shall be a certified material. One solid LCS shall be prepared and analyzed for every group of solid samples in a Sample Delivery Group, or for each batch of samples digested and/or distilled, whichever is more frequent.

5.9.6 Determine the concentration of each analyte in the LCS and calculate the percent recovery using Equation 1. The percent recovery control

limits for the LCS are 80-120%, inclusive (exception: the control limits for Ag and Sb are 50-120%). These limits must be used unless, for a solid LCS, other limits are provided by the LCS supplier. A copy of the supplier provided limits and true values must be included with the raw data.

5.9.7 If the results for the LCS analysis fall outside the control limits, the analyses shall be terminated, the problem corrected, and the samples associated with that LCS redigested and reanalyzed.

5.9.8 All LCS results, the measured concentrations, the true values, and the percent recoveries (%R), must be reported on FORM VII as indicated in Exhibit B.

5.9.9 LCS criteria must be met before any samples, QC samples, or required blanks are analyzed. Any samples, QC samples or required blanks analyzed when the LCS criteria have not been met must be redigested and reanalyzed at no additional cost to the Agency. Reanalyses must be performed within contract required holding times and must meet all sample acceptance criteria.

5.9.10 Sample results reported with a non-compliant LCS after reanalysis shall receive a commensurate reduction in sample price or zero payment due to data rejection depending upon the impact of the non-compliance on data usability.

5.10 ICP Serial Dilution Analysis (L)

5.10.1 The presence of matrix interferences during ICP analyses is verified with a serial dilution analysis. A serial dilution sample is prepared by diluting a aliquot of prepared sample by a factor of five (e.g., diluting 10.0 mL of prepared sample to 50.0 mL). The diluent shall be reagent water with the same acid content as the calibration standards. The ICP Serial Dilution Analysis shall be performed on a sample from each group of samples of the same matrix (i.e., aqueous, soil) and concentration (i.e., low, medium) or for each Sample Delivery Group, whichever is more frequent.

5.10.1.1 Samples identified as equipment or trip blanks shall not be used for Serial Dilution Analysis.

5.10.1.2 Unless otherwise specified, the same field samples shall be used for the serial dilution, matrix spike and duplicate analyses.

5.10.2 For each analyte in the serial dilution sample with a concentration of at least 50 times the MDL, calculate the percent differences using Equation 5.

EQ. 5

$$\% \, Difference = \frac{|I - S|}{I} \times 100$$

where,

I = Initial Sample Result
S = Serial Dilution Result (corrected for the 5x dilution)

5.10.3 The serial dilution result (corrected for the five fold dilution) must then agree within 10% of the original determination. If the dilution analysis for one or more analytes is not at or within 10%, a chemical or physical interference effect must be suspected, and the data for all affected analytes in the samples received associated with that serial dilution shall be flagged with an "E" on FORM IX and FORM I.

5.10.4 When there is more than one serial dilution per SDG, and one serial dilution result is not within contract criteria, flag all samples of the of the same matrix and concentration for that analyte in the Sample Delivery Group. Serial dilution results and "E" flags shall be reported on FORM IX.

5.10.5 ICP serial dilutions must be prepared and analyzed along with the samples, QC samples, and required blanks in an SDG. If required ICP serial dilutions are not prepared and analyzed during the original analytical run sequence, the contractor shall prepare and analyze the serial dilutions along with the original samples, during a valid analytical sequence, at no additional cost to the Agency. Reanalyses must be performed within contract required holding times and must meet all sample acceptance criteria.

5.10.6 Sample results reported without serial dilution analyses shall receive a commensurate reduction in sample price depending upon the impact of the non-compliance on data usability.

5.11 SDG-Specific Performance Evaluation (PE) Samples

5.11.1 Summary of SDG-Specific PE Samples

The Region I Performance Evaluation (PE) program has two functions: (1) to evaluate laboratory operation and protocols over a period of time, and (2) to provide information on the quality of individual data packages.

5.11.2 Frequency of SDG-Specific PE Samples

5.11.2.1 The Region shall submit PE samples with every SDG per parameter, matrix and concentration level (as available). The Region may obtain these SDG-Specific PE samples from either a commercial vendor or from the CLP National Program Office (NPO) which provides PE samples in support of the Superfund program. PE samples provided by the CLP-NPO are referred to as "EPA generated". PE samples recieved within a Case shall be assigned to an SDG containing field samples for that Case.

5.11.2.2 When the Region submits equipment blanks and/or Performance Evaluation samples (PEs) with soil/sediment/solid field samples, then the Contractor shall not perform a spike, duplicate, or serial dilution sample analysis on the aqueous matrix (equipment blank, PE sample). When the Region submits an aqueous PE sample with aqueous field samples, then the Contractor shall not choose the PE sample for the spike, duplicate, or serial dilution sample analysis.

5.11.2.3 If the PE sample is received as an ampulated standard, the ampulated PE sample is not considered to be another matrix type.

5.11.3 Procedure for Preparing SDG-Specific PE Samples

5.11.3.1 Instructions for preparation of the PE samples will be included with each submission of PE samples.

5.11.3.2 If PE sample instructions do not apply to the PE sample received, then the Contractor must contact the RSCC to ascertain whether or not to analyze the PE sample and/or to obtain appropriate PE sample instructions or another PE sample.

5.11.4 Calculations for SDG-Specific PE Samples

For EPA-generated and commercially prepared PE samples that are submitted with each SDG, the Contractor must correctly identify and quantitate all Target Analyte List (TAL) analytes using the method specific criteria presented in Exhibits D and E.

5.11.5 Technical Acceptance Criteria for SDG-Specific PE Samples

5.11.5.1 All SDG-Specific PE samples must be analyzed under the same analytical conditions as the associated analytical samples and must meet the same QA/QC acceptance criteria specified in this Exhibit as that established for sample analyses.

5.11.5.2 EPA-generated PE samples included with the SDG shall be evaluated by the Region using a CLP NPO computer program called PeacTOOLs. PeacTOOLs rates the PE sample results based on statistically generated confidence intervals.

5.11.5.3 The results of commercially prepared PE samples will be evaluated using the vendors' statistically generated confidence intervals.

5.11.5.4 Contractor results for the SDG-Specific PE samples will be evaluated using the most recent Region I data validation criteria for PE samples.

5.11.5.5 At a minimum, the PE results will be evaluated for analyte identification, quantitation, and sample contamination. Confidence intervals for the quantitation of target analytes are based on reported values using population statistics. The Agency may adjust the criteria on any given PE sample to compensate for unanticipated difficulties with a particular sample.

5.11.6 Corrective Action for SDG-Specific PE Samples

5.11.6.1 If the technical acceptance criteria specified in this Exhibit for field samples are not met for the PE sample results, the same corrective actions which are established for sample analysis must also be followed for the PE sample.

5.11.6.2 If an SDG-Specific PE sample evaluated by Region I as described in Section 5.11.5 above, indicates unacceptable laboratory performance, then the Contractor may be required to reanalyze all samples, standards, blanks and QC samples associated with the unacceptable PE sample result (if sufficient volume remains) and/or analyze a new PE sample at no additional cost to the Agency. Unacceptable laboratory performance includes either a TAL false positive result, false negative result, and/or analyte misquantitation.

5.11.6.3 SDG-Specific sample results reported with unacceptable PE results shall be subject to a commensurate reduction in sample price or zero payment due to data rejection, depending upon the impact of the non-compliance on data usability.

5.12 Method Detection Limit (MDL) Determination

5.12.1 The method detection limits (MDLs) shall be determined at least annually (15th of January), as required in Exhibit B, for each target analyte, matrix, method and instrument used for this Contract and shall meet the CRQL requirements specified in Exhibit C. The MDL values must be less than or equal to one-third of the CRQLs. MDL studies must be performed in accordance with 40 Code of Federal Regulations, Part 136, Appendix B and this conract for the following target analytes and matrices: Medium Level Metals, aqueous; Medium Level Metals, solid; Medium Level Cyanide, aqueous; Medium Level Cyanide, solid; Low Level Metals, aqueous; Low Level Cyanide, aqueous; Total Organic Carbon, aqueous; and Total Organic Carbon, solid.

5.12.2 The MDL determinations must be conducted using the same QA/QC specifications as for sample analysis. These specifications include, but are not limited to criteria for: initial calibration, ICV/ICB, CCV/CCB, PB, and LFB. All QC requirements for frequency, acceptance criteria, and corrective actions must be achieved. Documentation for MDL studies must include Forms 2A, 2B, 3, 10, 13, and 14. Supporting raw data documentation must identify the source of the standards, concentration of the standards, all preparation volumes and weights, and the sequence of analysis. The deliverable must clearly demonstrate that an appropriate analyte and interference free blank matrix was utilized as specified in 40 CFR, Part 136, Appendix B.

5.12.3 For each instrument and method, MDLs for all analytes in Exhibit C must be established in accordance with 40 CFR, Part 136, Appendix B. A minimum of seven aliquots of appropriate blank matrix (reagent water or blank sand/soil) spiked at 1-5 times the estimated MDL must be taken through the entire sample preparation and analysis procedure. If more than seven aliquots are analyzed, then they must also be included in the MDL values calculation. All sequential analyses of MDL standards must be reported and used in the resulting MDL values which are calculated. The MDL studies are calculated as described in 40 CFR, Part 136, Appendix B and reported as a separate SDG in accordance with Exhibit B. The appropriate Students' t value must be clearly provided with the algorithm used to calculate the MDL values. MDLs shall be determined and reported for each method and wavelength used in the analysis of the samples.

5.12.4 The MDL study must be reported as detailed in Exhibit B. The individual analytical run raw data must be provided and these data must be summarized in a table which demonstrates the calculated MDL values. The summarized MDL results table must include the concentration found for each analyte in each aliquot, the mean concentration of each analyte, the percent recovery of each analyte, the standard deviation for each analyte, and the Method Detection Limit. The true concentration of the analyte in the spike solution must also be provided. The table must list the analytes in the same order as they appear in the target analyte list in Exhibit C. In addition, MDLs shall be reported on Forms XA, XB, and XC for each instrument and method used in reporting results for an SDG and shall be submitted with each data package. Forms 2A, 2B, 3, 10A, 10B, 10C, 13, and 14 must be provided in the MDL deliverable.

Note: The MDL for TOC analysis in soil/sediment samples shall be determined in total mg carbon and shall specify, in the "Comments"

section on FORM X - PART 2, the minimum dry weight of sample that must be analyzed in order to meet the 100 mg/Kg CRQL.

5.12.4 The annually determined MDL for an instrument and method shall always be used as the MDL for that instrument/method during that year. If the instrument/method is adjusted in any way that may affect the MDL, the MDL for that instrument/method must be redetermined and the results submitted for use as the established MDL for that instrument/method for the remainder of the year.

5.13 Interelement Corrections for ICP (IEC)

5.13.1 The ICP interelement correction factors shall be determined at least semiannually (i.e., 15th of January and July). The IEC determinations must be conducted using the same specifications as for sample analyses. These specifications include, but are not limited to: ICV/ICB criteria, LFB criteria, and CCV/CCB criteria. Correction factors for spectral interference due to Al, Ca, Fe, and Mg shall be determined and reported for all ICP instruments at all wavelengths used for each analyte reported by ICP. Correction factors for spectral interference due to analytes other than Al, Ca, Fe, and Mg shall also be reported. In addition, IECs shall be reported on Form XI for each ICP instrument used in reporting ICP results for an SDG and shall be submitted with each data package as specified in Exhibit B.

5.13.2 If the instrument was adjusted in any way that may affect the ICP interelement correction factors, the factors shall be redetermined and the results submitted for use. In addition, all data used for the determination of the interelement correction factors shall be available to the USEPA during an on-site laboratory evaluation.

5.14 Linear Range Analysis (LRA)

5.14.1 The linear range of each analyte analyzed by ICP shall be determined at least semiannually (i.e., 15th of January and July). The linear range verification check standards must be analyzed using the same specifications as for sample analyses. These specifications include, but are not limited to: ICV/ICB criteria, LFB criteria, ICS criteria, and CCV/CCB criteria. The analytically determined concentration of this standard must be within 5% of the true value. This concentration is the upper limit of the ICP linear range beyond which results shall not be reported under this contract without dilution of the analytical sample. The linear range verification check standards shall be reported on Form XII for each instrument and wavelength used in reporting results for an SDG and shall be submitted with each data package.

5.14.2 If the instrument was adjusted in any way that may affect the ICP linear range, the linear range verification check standards shall be redetermined and the results submitted for use. In addition, all data used for the determination of the analyte linear ranges shall be available to the USEPA during an on-site laboratory evaluation.

5.15 Furnace Atomic Absorption (AA) QC Analyses

5.15.1 The nature of the Furnace AA technique requires the procedures summarized in Figure 1, Furnace AA Analysis Scheme ("MSA Tree"), for quantitation. (These procedures do not replace those in Exhibit D of this SOW, but supplement the guidance provided therein.)

5.15.2 All furnace analyses results shall be within the calibration range. In addition, all analyses (except during full method of standard additions (MSA)) shall require a minimum of duplicate injections.

5.15.2.1 The absorbance or concentration of each injection shall be reported in the raw data as well as the average absorbance or concentration values and the relative standard deviation (RSD) or coefficient of variation (CV). Average concentration values are used for reporting purposes. The Contractor shall be consistent per method and SDG in choosing absorbance or concentration to evaluate which route is to be followed in the MSA Tree. The Contractor shall also indicate which of the two is being used if both absorbance and concentration are reported in the raw data.

5.15.2.2 For MSA analysis, the absorbance of each injection shall be included in the raw data. A maximum of 10 full sample analyses, to a maximum 20 injections, may be performed between continuing calibration verification and continuing calibration blank analyses.

5.15.2.3 For concentrations greater than the CRQL, the duplicate injection readings must agree within 20% RSD or CV, or the analytical sample shall be rerun once (i.e., two additional burns). If the readings are still out, flag the value reported on FORM I with an "M". The "M" flag is required for the analytical spike as well as the sample. If the analytical spike for a sample requires an "M" flag, the flag shall be reported on FORM I for that sample.

5.15.3 All furnace analyses for each analytical sample, including those requiring an "M" flag, shall require at least an analytical spike to determine if the MSA shall be required for quantitation.

5.15.3.1 The analytical spike[4] shall be required to be at a concentration (in the sample) of 2x CRQL (except for lead which must be at 20 ug/L).

5.15.3.1.1 Low Level Aqueous Samples

For all target analytes in Low Level aqueous samples (whether preconcentration of the sample was required or not), the analytical spikes, in the final sample digestate ready for analysis, shall be at concentrations equal to the following: Sb at 30 µg/L; As, Cr, Cu, Pb, Ni, and Tl at 20 µg/L; Be, Se, and Ag at 10 µg/L; and Cd at 2 µg/L.

[4]Analytical spikes are furnace spikes to be prepared prior to analysis, but after digestion (if performed), by adding a known quantity of the analyte to an aliquot of the sample. The unspiked sample aliquot shall be compensated for any volume change in the spike samples by the addition of deionized water to the unspiked sample aliquot. The volume of the spiking solution added shall not exceed 10% of the analytical sample volume; this requirement also applies to MSA spikes.

5.15.3.2 The requirement for an analytical spike shall include the LCS, LFB, and the preparation blank. (The LCS and LFB shall be quantitated from the calibration curve and corrective action, if needed, taken accordingly. MSA is not to be performed on the LCS, LFB, or preparation blank, regardless of the analytical spike recovery results.) If the preparation blank analytical spike recovery is out of control (85-115%), the spiking solution shall be verified by respiking and rerunning the preparation blank once. If the preparation blank analytical spike recovery is still out of control, correct the problem and reanalyze all analytical samples associated with that blank.

5.15.3.3 An analytical spike shall not be performed on the matrix spike sample.

5.15.3.4 The analytical spike of a sample shall be run immediately after that sample. The percent recovery (%R) of the spike, calculated by the same formula as Spike Sample Analyses (see Equation 3), shall then determine how the sample shall be quantitated, as follows:

5.15.3.4.1 If the spike recovery is less than 40%, the sample shall be diluted and rerun with another spike. Dilute the sample by a factor of 5 to 10 and rerun. This step shall only be performed once. If after the dilution the spike recovery is still <40%, report data and flag with an "E" to indicate interference problems.

5.15.3.4.2 If the spike recovery is greater than or equal to 40% and the sample absorbance or concentration is less than 50% of the "spike"[5], report the sample results to the MDL. If the spike recovery is less than 85% or greater than 115%, flag the result with a "W".

5.15.3.4.3 If the sample absorbance or concentration is greater than or equal to 50% of the "spike"[7] and the spike recovery is at or between 85% and 115%, the sample shall be quantitated directly from the calibration curve and reported down to the MDL.

5.15.3.4.4 If the sample absorbance or concentration is greater than or equal to 50% of the "spike"[7] and the spike recovery is less than 85% or greater than 115%, the sample shall be quantitated by MSA.

5.15.4 The following procedures shall be incorporated into MSA analyses.

5.15.4.1 Data from MSA calculations shall be within the linear range as determined by the calibration curve generated at the beginning of the analytical run.

5.15.4.2 The sample and three spikes shall be analyzed consecutively (MS0, MS1, MS2, MS3) for MSA quantitation (the "initial" spike run data are specifically excluded from use in the MSA quantitation). Only single injections shall be performed for MSA quantitation.

5.15.4.2.1 Each full MSA counts as two analytical samples towards determining 10% QC frequency (i.e., five full MSAs can be performed between calibration verifications).

5.15.4.3 For analytical runs containing only MSAs, single injections can be used for QC samples during that run. For instruments that operate in an MSA mode only, MSA can be used to determine QC samples during that run.

5.15.4.4 Spikes shall be prepared such that:

a) Spike 1 is approximately 50% of the apparent sample concentration (direct instrument reading).

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[5]"Spike" is defined as [absorbance or concentration of spike sample] minus [absorbance or concentration of the sample].

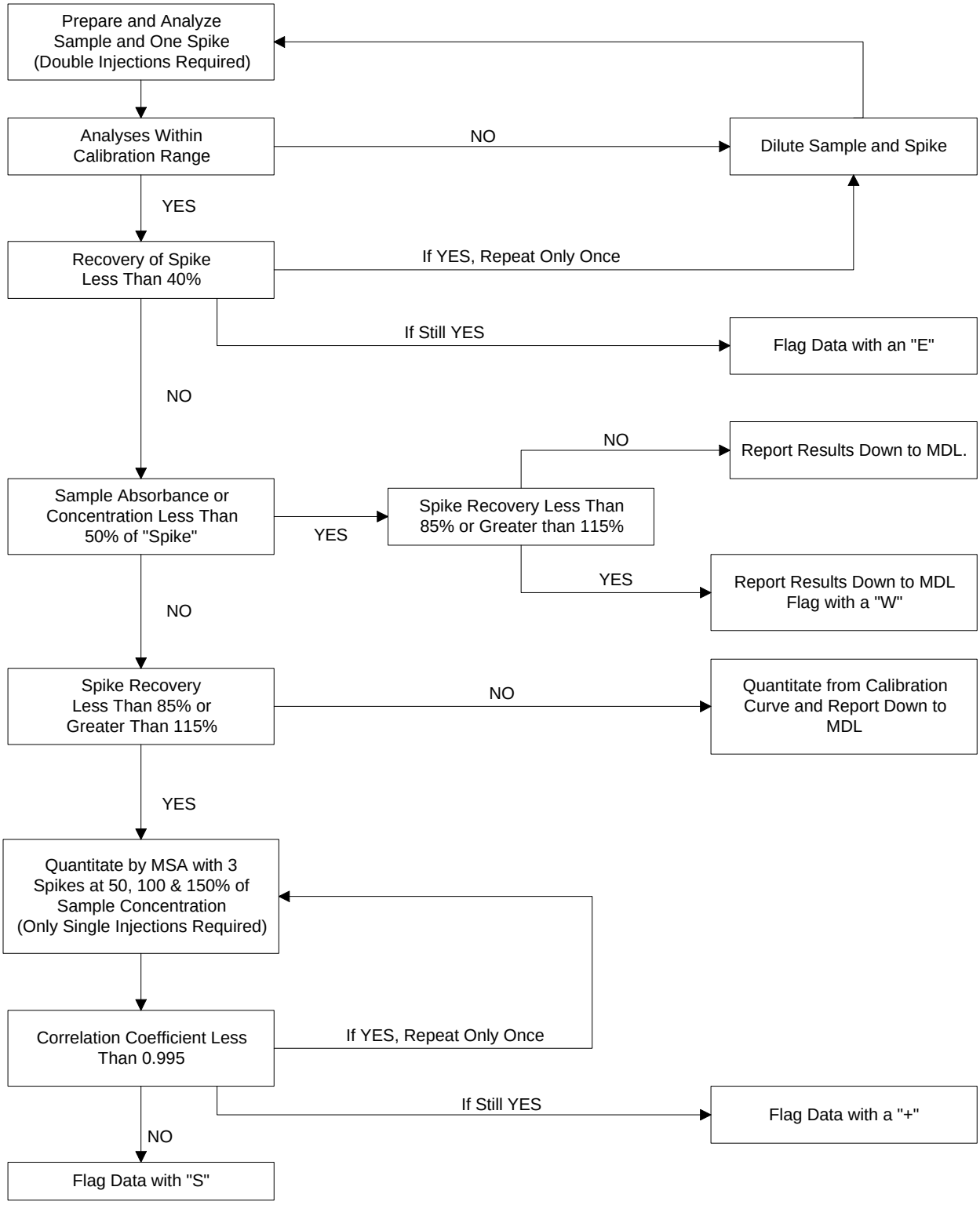
b) Spike 2 is approximately 100% of the apparent sample concentration.

c) Spike 3 is approximately 150% of the apparent sample concentration.

5.15.4.5 The data for each MSA analysis shall be clearly identified in the raw data documentation (using added concentration as the x-variable and absorbance as the y-variable) along with the slope, x-intercept, y-intercept and correlation coefficient (r) for the least squares fit of the data. The MSA results shall be reported on FORM VIII. Reported values obtained by MSA shall be flagged on the data sheet (FORM I) with the letter "S" if the correlation coefficient is greater than or equal to 0.995.

5.15.4.6 If the correlation coefficient (r) for a particular analysis is less than 0.995, the MSA analysis shall be repeated once. If the correlation coefficient is still less than 0.995, report the results on FORM I from the run with the best "r" and flag the result with a "+" on FORM VIII and FORM I.

FIGURE 1. FURNACE ATOMIC ABSORPTION ANALYSIS SCHEME



5.16 Tables

TABLE 1

INITIAL AND CONTINUING CALIBRATION VERIFICATION
CONTROL LIMITS FOR INORGANIC ANALYSES

		% of True Value	
Analytical Method	Inorganic Species	Low Limit	High Limit
ICP/FAA/GFAA	Metals	90	110
Cold Vapor AA	Mercury	80	120
Other	Cyanide, TOC	85	115

TABLE 2

INTERFERENT AND ANALYTE ELEMENTAL CONCENTRATIONS
USED FOR ICP INTERFERENCE CHECK SAMPLE (ICS)

Analytes	(mg/L)	Interferents	(mg/L)
Ag	0.2	Al	500
As	0.1	Ca	500
Ba	0.5	Fe	200
Be	0.5	Mg	500
Cd	1.0		
Co	0.5		
Cr	0.5		
Cu	0.5		
Mn	0.5		
Ni	1.0		
Pb	0.05		
Sb	0.6		
Se	0.05		
Tl	0.1		
V	0.5		
Zn	1.0		

TABLE 3

MEDIUM LEVEL SPIKING LEVELS FOR SPIKE SAMPLE ANALYSIS

	For ICP/AA		For Furnace AA[4]		Other[1][2]
Element	Aqueous (ug/L)	Soil[2] (mg/Kg)	Aqueous (ug/L)	Soil[2] (mg/Kg)	
Aluminum	2,000	*			
Antimony	500	100	100	20	
Arsenic	2,000	400	40	8	
Barium	2,000	400			
Beryllium	50	10	10	2	
Cadmium	50	10	5	1	
Calcium	*	*			
Chromium	200	40	40	8	
Cobalt	500	100			
Copper	250	50	40	8	
Iron	1,000	*			
Lead	500	100	20	4	
Magnesium	*	*			
Manganese	500	100			
Mercury					1
Nickel	500	100	25	5	
Potassium	*	*			
Selenium	2,000	400	10	2	
Silver	50	10	20	4	
Sodium	*	*			
Thallium	2,000	400	50	10	
Vanadium	500	100			
Zinc	500	100			
TOC					1,000
Cyanide					100 ug/L[3]

* No spike required. **NOTE**: Elements without spike levels, and not designated with an asterisk, shall be spiked at appropriate levels.

(1) Specified spiking levels are for both aqueous and soil/sediment matrices. Reporting units are ug/L and mg/kg, respectively.

(2) The levels shown indicate concentrations in the final solution of the spiked sample (100 mL for mercury and 200 mL for all other metals) when the wet weight of 1 gram (for ICP, Furnace AA, and Flame AA), or 0.2 grams (for mercury), of sample is taken for analysis. Adjustment shall be made to maintain these spiking levels when the weight of sample taken deviates by more than 10% of these values. Appropriate adjustment shall be made for microwave digestion procedures where 0.5 grams of sample or 50.0 mL (45.0 mL of sample plus 5.0 mL of acid) of aqueous sample are required for analysis.

(3) The level shown indicates the cyanide concentration in the final sample solution prepared for analysis (i.e., post-distillation). The final volume of the sample after distillation shall be the basis for the amount of cyanide to be added as the spike. For instance, the full volume distillation procedure shall require the addition of 25 ug cyanide to the sample prior to distillation (based on the final

distillate volume of 250 mL) to meet the specified spiking level; and the midi distillation procedure requires the addition of 5 ug of cyanide to the sample prior to distillation (based on the final distillate volume of 50 mL).

For soil samples, the final sample solution prepared for analysis (i.e., the distillate) must contain cyanide spiked at a concentration of 100 ug/L regardless of the distillation procedure employed or the amount of sample used for distillation. Use the final sample volume after distillation as the basis for the amount of cyanide to add as the spike. The units for reporting soil/solid sample cyanide results shall be mg/kg. To convert from ug/L to mg/kg, use the equation below:

$$mg/kg = ug/L \times \frac{final\ distillate\ volume\ (L)}{sample\ weight\ (g)}$$

(4) If the Contractor uses an Inductively Coupled Plasma (ICP) spectrometer to analyze field samples for those elements for which Furnace AA spikes are present in Table 3 (e.g., Sb, As, Be, Cd, Cr, Cu, Ni, Pb, Se, Ag, and/or Tl), the spiking concentrations shown for furnace AA analyses (Table 3) shall also apply to the ICP analysis for those elements, provided the ICP MDLs for those elements meet the CRQL requirements in Exhibit C. Otherwise, those elements shall be spiked at the ICP levels specified in Table 3.

TABLE 4

SPIKING LEVELS FOR LOW LEVEL AQUEOUS SAMPLES AND LABORATORY CONTROL SAMPLE[1]

Element	ICP µg/L	Furnace AA µg/L	Other µg/L
Aluminum	1000		
Antimony	200	50	
Arsenic	200	20	
Barium	200		
Beryllium	25	5	
Cadmium	25	3	
Calcium	*		
Chromium	100	20	
Cobalt	200		
Copper	100	20	
Iron	500		
Lead	100	10	
Magnesium	*		
Manganese	200		
Mercury			0.5
Nickel	200	20	
Potassium	*		
Selenium	100	5	
Silver	25	10	
Sodium	*		
Thallium	100	25	
Vanadium	200		
Zinc	200		
Cyanide			50[2]

* No spike required.

(1) Levels shown indicate concentrations in the original sample (prior to digestion).

(2) The level shown indicates the cyanide concentration in the final sample solution prepared for analysis (i.e., post-distillation). The final volume of the sample after distillation shall be the basis for the amount of cyanide to be added as the spike. For instance, the full volume distillation procedure shall require the addition of 12.5 µg cyanide to the sample prior to distillation (based on the final distillate volume of 250 mL) to meet the specified spiking level.

(4) If the Contractor uses an Inductively Coupled Plasma (ICP) spectrometer to analyze field samples for those elements for which Furnace AA spikes are present in Table 4 (e.g., Sb, As, Be, Cd, Cr, Cu, Ni, Pb, Se, Ag, and/or Tl), the spiking concentrations shown for furnace AA analyses (Table 4) shall also apply to the ICP analysis for those elements, provided the ICP MDLs for those elements meet the CRQL requirements in Exhibit C. Otherwise, those elements shall be spiked at the ICP levels specified in Table 4.

6.0 ANALYTICAL STANDARD REQUIREMENTS

The U.S. Environmental Protection Agency may be unable to supply analytical reference standards either for direct analytical measurements or for the purpose of traceability. In these cases, all contract laboratories will be required to prepare from materials or purchase from private chemical supply houses those standards necessary to successfully and accurately perform the analyses required in this protocol.

6.1 Preparation of Chemical Standards from the Neat High Purity Bulk Material

If the laboratory cannot obtain analytical reference data from the U.S. EPA, the laboratory may prepare their own chemical standards. Laboratories shall obtain the highest purity possible when purchasing chemical standards; standards purchased at less than 97% purity shall be documented as to why a higher purity could not be obtained.

6.1.1 If required by the manufacturer, the chemical standards shall be kept refrigerated when not being used in the preparation of standard solutions. Proper storage of chemicals is essential in order to safeguard them from decomposition.

6.1.2 The purity of a compound can sometimes be misrepresented by a chemical supply house. Since knowledge of purity is needed to calculate the concentration of solute in a solution standard, it is the contract laboratory's responsibility to have analytical documentation ascertaining that the purity of each compound is correctly stated. Purity confirmation, when performed, should use appropriate techniques. Use of two or more independent methods is recommended. The correction factor for impurity when weighing neat materials in the preparation of solution standards is:

Equation 1

$$\textit{weight of impure compound} = \frac{\textit{weight of pure compound}}{(\textit{percent purity}/100)}$$

where "weight of pure compound" is that required to prepare a specific volume of a solution standard of a specified concentration.

6.1.3 Mis-identification of compounds occasionally occurs and it is possible that a mislabeled compound may be received from a chemical supply house. It is the contract laboratory's responsibility to have analytical documentation ascertaining that all compounds used in the preparation of solution standards are correctly identified.

6.1.4 Log notebooks are to be kept for all weighing and dilutions. All subsequent dilutions from the primary standard and the calculations for determining their concentrations are to be recorded and verified by a second person. All solution standards are to be refrigerated, if required, when not in use. All solution standards are to be clearly labeled as to the identity of the analyte or analytes, concentration, date prepared, solvent, and initials of the preparer.

6.2 Purchase of chemical standards already in solution

6.2.1 Solutions of analytical reference standards can be purchased by Contractors provided they meet the following criteria:

Laboratories shall maintain documentation of the purity confirmation of the material to verify the integrity of the standard solutions they purchase.

6.2.2 The Contractor shall purchase standards for which the quality is demonstrated statistically and analytically by a method of the supplier's choice. One way this can be demonstrated is to prepare and analyze three solutions; a high standard, a low standard, and a standard at the target concentration (see parts a and b below). The supplier must then demonstrate that the analytical results for the high standard and low standard are consistent with the difference in theoretical concentrations. This is done by the Student's t-test in part "d". If this is achieved, the supplier must then demonstrate that the concentration of the target standard lies midway between the concentrations of the low and high standards. This is done by the Student's t-test in part e. Thus the standard is certified to be within 10 percent of the target concentration.

If the procedure above is used, the supplier must document that the following have been achieved:

6.2.2.1 Two solutions of identical concentration shall be prepared independently from neat materials. An aliquot of the first solution shall be diluted to the intended concentration (the "target standard"). One aliquot is taken from the second solution and diluted to a concentration ten percent greater than the target standard. This is called the "high standard". One further aliquot is taken from the second solution and diluted to a concentration 10 percent less than the target standard. This is called the "low standard".

6.2.2.2 Six replicate analyses of each standard (a total of 18 analyses) shall be performed in the following sequence: low standard, target standard, high standard, low standard, target standard, high standard, ...

6.2.2.3 The mean and variance of the six results for each solution shall be calculated.

Equation 2

$$MEAN = \frac{Y_1 + Y_2 + Y_3 + Y_4 + Y_5 + Y_6}{6}$$

Equation 3

$$VARIANCE = \frac{Y_1^2 + Y_2^2 + Y_3^2 + Y_4^2 + Y_5^2 + Y_6^2 - (6 * MEAN)^2}{5}$$

The values Y_1, Y_2, Y_3, ..., represent the results of the six analyses of each standard. The means of the low, target, and high standards are designated M_1, M_2, and M_3, respectively. The variances of the low, target, and high standards are designated V_1, V_2, and V_3, respectively. Additionally, a pooled variance, V_p, is calculated.

Equation 4

$$V_p = \frac{\frac{V_1}{0.81} + V_2 + \frac{V_3}{1.21}}{3}$$

If the square root of V_p is less than one percent of M_2, then $M_2^2/10{,}000$ is to be used as the value of V_p in all subsequent calculations.

6.2.2.4 The test statistic shall be calculated:

Equation 5

$$TEST\ STATISTIC = \frac{\left| \frac{M_3}{1.1} - \frac{M_1}{0.9} \right|}{\left(\frac{V_p}{3} \right)^{0.5}}$$

If the test statistic exceeds 2.13, then the supplier has failed to demonstrate a twenty percent difference between the high and low standards. In such a case, the standards are not acceptable.

6.2.2.5 The test statistic shall be calculated:

Equation 6

$$TEST\ STATISTIC = \frac{\left| M_2 - \left(\frac{M_1}{1.8} \right) - \left(\frac{M_3}{2.2} \right) \right|}{\left(\frac{V_p}{4} \right)^{0.5}}$$

If the test statistic exceeds 2.13, the supplier has failed to demonstrate that the target standard concentration is midway between the high and low standards. In such a case, the standards are not acceptable.

6.2.2.6 The 95 percent confidence intervals for the mean result of each standard shall be calculated:

Equation 7

$$Interval\ for\ Low\ Standard = M_1 \pm 2.13 \left(\frac{V_p}{6} \right)^{0.5}$$

Equation 8

$$Interval\ for\ Target\ Standard = M_2 \pm 2.13 \left(\frac{V_p}{6} \right)^{0.5}$$

Equation 9

$$\textit{Interval for High Standard} = M_3 \pm 2.13 \left(\frac{V_p}{6} \right)^{0.5}$$

These intervals shall not overlap. If overlap is observed, then the supplier has failed to demonstrate the ability to discriminate the 10 percent difference in concentrations. In such a case, the standards are not acceptable. In any event, the laboratory is responsible for the quality of the standards employed for analyses under this contract.

6.3 Requesting Standards From the EPA Standards Repository

Solutions of analytical reference materials can be ordered from the U.S. EPA Chemical Standards Repository, **depending on availability**. The Contractor can place an order for standards only after demonstrating that these standards are not available from commercial vendors either in solution or as a neat material.

6.4 Documentation of the Verification and Preparation of Chemical Standards

It is the responsibility of each laboratory to maintain the necessary documentation to show that the chemical standards they have used in the performance of REAP analyses conform to the requirements previously listed. Weighing logbooks, calculations, raw data, etc., whether produced by the laboratory or purchased from chemical supply houses, shall be maintained by the laboratory and may be subject to review during on-site inspection visits. In those cases where the documentation is supportive of the analytical results of data packages sent to EPA, such documentation is to be kept on file by the laboratories and submitted to EPA upon completion of the contract.

6.4.1 Upon request by the EPA, the Contractor shall submit their most recent previous year's documentation (12 months) for the verification and preparation of chemical standards within 14 days of the receipt of request to the recipients he/she designates.

6.4.2 The Agency may generate a report discussing deficiencies in the Contractor's documentation for the verification and preparation of chemical standards or may discuss the deficiencies during an on-site laboratory evaluation. In a detailed letter to the EPA, the Contractor shall address the deficiencies and the subsequent corrective action implemented by the Contractor to correct the deficiencies within 14 days of receipt of the report or the on-site laboratory evaluation. An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of the Agency to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the EPA why he/she is unable to meet the delivery schedule listed in this section. The EPA will not grant an extension for greater than 14 days for the Contractor's response letter to the standards documentation report. The Contractor shall proceed and not assume that an extension will be granted until so notified by the EPA.

6.4.3 If new SOPs are required to be written, or if existing SOPs are required to be rewritten or amended because of deficiencies and subsequent corrective action implemented by the Contractor, the Contractor shall write/amend the SOPs per the requirements listed in Exhibit E, Section IV.

6.4.4 If the Contractor fails to adhere to the requirements listed in this Section, a Contractor may expect, but the Agency is not limited to the following actions: reduction in the number of samples sent under the contract, suspension of sample shipment to Contractor, data package audit, an on-site laboratory evaluation, a remedial laboratory evaluation sample, and/or contract sanctions, such as a Cure Notice.

7.0 DATA PACKAGE AUDITS

7.1 Overview

Data package audits are performed by the Agency for program overview and specific Regional concerns. Standardized procedures have been established to assure uniformity of the auditing process. Data packages are periodically selected from recently received Cases. They are evaluated for the technical quality of hardcopy raw data, quality assurance, and the adherence to contractual requirements. This function provides external monitoring of program QC requirements.

Data package audits are used to assess the technical quality of the data and evaluate overall laboratory performance. Audits provide the Agency with an in-depth inspection and evaluation of the Case data package with regard to achieving QA/QC acceptability. A thorough review of the raw data is completed including: all instrument readouts used for the sample results, instrument printouts, and other documentation for deviations from the contractual requirements, a check for transcription and calculation errors, a review of the qualifications of the laboratory personnel involved with the Case, and a review of all current SOPs on file.

7.2 Responding to the Data Package Audit Report

After completion of the data package audit, the Agency may send a copy of the data package audit report to the Contractor or may discuss the data package audit report on an on-site laboratory evaluation. In a detailed letter to the EPA, the Contractor shall discuss the corrective actions implemented to resolve the deficiencies listed in the data package audit report within 14 days of receipt of the report. An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of the Agency to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the EPA why he/she is unable to meet the delivery schedule listed in this section. The EPA will not grant an extension for greater than 14 days for the Contractor's response letter to the data package report. The Contractor shall proceed and not assume that an extension will be granted until so notified by the EPA.

7.2.1

If new SOPs are required to be written, or if existing SOPs are required to be rewritten or amended because of deficiencies and subsequent corrective action implemented by the Contractor, the Contractor shall write/amend the SOPs per the requirements listed in Exhibit E, Section IV.

7.3 Corrective Action

If the Contractor fails to adhere to the requirements listed in this section, the Contractor may expect, but the Agency is not limited to the following actions: reduction in the number of samples sent under the contract, suspension of sample shipment to the Contractor, an on-site laboratory evaluation, data package audit, remedial performance evaluation sample, and/or contract sanctions, such as a Cure Notice.

7.4 Regional Data Review

Contractor data are generated to meet the specific needs of EPA New England. In order to verify the useability of data for the intended purpose, the Region reviews data from the perspective of the end user, based on functional guidelines for data review which have been developed jointly by the Regions and the National Program Office. EPA New England uses these guidelines as the basis for data evaluation. The Region may augment the basic guideline review process with additional review based on Region-specific or site-specific concerns. Regional reviews, like the sites under investigation, vary based on the nature of the problem under investigation and the Regional response appropriate to the specific circumstances.

Regional data reviews, relating usability of the data to a specific site, are part of the collective assessment process which includes reviews designed to identify contractual discrepancies. These different types of evaluations are integrated into a collective review that is

necessary for Program and Contractor administration and management and may be used to take appropriate action to correct deficiencies in the Contractor's performance.

8.0 QUARTERLY BLIND (QB) LABORATORY EVALUATION PROGRAM

8.1 Summary of QB Samples

The Region will also submit quarterly laboratory evaluation samples for specified analyses in conjunction with the CLP Quarterly Blind (QB) program. The results from the analysis of these QB samples will be used by the Region to verify the Contractor's continuing ability to produce acceptable analytical data. The results will also be used to assess the precision and accuracy of the analytical methods for specific analytes.

8.2 Frequency of CLP QB Samples

The Region will submit laboratory evaluation samples on a quarterly basis for specified analyses in conjunction with the CLP Quarterly Blind (QB) program.

8.3 Procedure for Preparing CLP QB Samples

Instructions for preparation of the QB samples will be included with each submission of QB samples.

8.4 Calculations for CLP QB Samples

The Contractor must correctly identify and quantitate all TAL analytes using the method specific criteria presented in Exhibits D and E.

8.5 Technical Acceptance Criteria for CLP QB Samples

8.5.1 The QB samples must be analyzed under the same analytical conditions and must meet the same acceptance criteria as established for sample analyses.

8.5.2 The QB samples will be scored and the results will be used to assess the precision and accuracy of the analytical methods for specific analytes.

8.5.3 At a minimum, the results are evaluated for analyte identification, quantitation, and sample contamination. Confidence intervals for the quantitation of target compounds are based on reported values using population statistics. The Agency may adjust the scores on any given laboratory evaluation sample to compensate for unanticipated difficulties with a particular sample.

8.5.4 The Contractor's performance on the QB samples will be measured and reported as follows:

8.5.4.1 Acceptable, No Response Required (Score greater than or equal to 90%): Data meets most or all of the scoring criteria.

8.5.4.2 Acceptable, Response Explaining Deficiency(ies) Required (Score greater than or equal to 75% but less than 90%): Deficiencies exist in the Contractor's performance.

8.5.4.3 Unacceptable Performance (Score less than 75%): Deficiencies exist in the Contractor's performance to the extent that the Agency has determined that the Contractor has not demonstrated the capability to meet the contract requirements.

8.5.4.4 In the case of Sections 8.5.4.2 and 8.5.4.3 above, the Contractor shall respond to the deficiency(ies) and the action(s) taken to correct the deficiency(ies) in a letter to the EPA within 14 days of receipt of notification from the Agency.

8.6 Corrective Action for CLP QB Samples

8.6.1 The corrective actions for QB sample results which do not meet the acceptance criteria defined in Section 8.5.1 above are the same corrective actions outlined for sample analysis in Section 5.0.

8.6.2 After receipt and review of the Contractor's deficiency letter (Section 8.5.4.4), the EPA shall notify the Contractor concerning the remedy for their unacceptable performance. The Contractor may expect, but the Agency is not limited to, the following actions: commensurate reduction in sample price, zero payment due to data rejection, reduction of the number of samples sent under the contract, suspension of sample shipment to the Contractor, a data package audit, an on-site laboratory evaluation, a remedial laboratory evaluation sample, and/or contract sanctions, such as a Cure Notice.

NOTE: The Contractor's prompt response demonstrating that corrective actions have been taken to ensure the Contractor's capability to meet contract requirements may facilitate continuation of sample scheduling.

9.0 ON-SITE LABORATORY EVALUATIONS

At a frequency dictated by a contract laboratory's performance, the EPA will conduct an on-site laboratory evaluation. On-site laboratory evaluations are carried out to monitor the Contractor's ability to meet selected terms and conditions specified in the contract. The evaluation process incorporates two separate categories: Quality Assurance Evaluation and an Evidentiary Audit.

9.1 Quality Assurance On-Site Evaluation

Quality assurance evaluators inspect the Contractor's facilities to verify the adequacy and maintenance of instrumentation, the continuity, experience and education of personnel, and the acceptable performance of analytical and QC procedures. The Contractor should expect that items to be monitored will include, but not be limited to, the following:

- Size and appearance of the facility
- Quantity, age, availability, scheduled maintenance and performance of instrumentation
- Availability, appropriateness, and utilization of the LQAP and SOPs
- Staff qualifications, experience, and personnel training programs
- Reagents, standards, and sample storage facilities
- Standard preparation logbooks and raw data
- Bench sheets and analytical logbook maintenance and review
- Review of the Contractor's sample analysis/data package inspection/data management procedures

Prior to an on-site evaluation, various documentation pertaining to performance of the specific Contractor is integrated in a profile package for discussion during the evaluation. Items that may be included are previous on-site reports, performance evaluation sample scores, Regional review of data, Regional QA materials, data audit reports, and data trend reports.

9.2 Evidentiary Audit

Evidence auditors conduct an on-site laboratory evaluation to determine if laboratory policies and procedures are in place to satisfy evidence handling requirements as stated in Exhibit F. The evidence audit is comprised of the following three activities:

9.2.1 Procedural Audit

The procedural audit consists of review and examination of actual standard operating procedures and accompanying documentation for the following laboratory operations: sample receiving, sample storage, sample identification, sample security, sample tracking (from receipt to completion of analysis) and analytical project file organization and assembly.

9.2.2 Written SOPs Audit

The written SOPs audit consists of review and examination of the written SOPs to determine if they are accurate and complete for the following laboratory operations: sample receiving, sample storage, sample identification, sample security, sample tracking (from receipt to completion of analysis) and analytical project file organization and assembly.

9.2.3 Analytical Project File Evidence Audit

The analytical project file evidence audit consists of review and examination of the analytical project file documentation. The auditors review the files to determine:

- The accuracy of the document inventory

- The completeness of the file
- The adequacy and accuracy of the document numbering system
- Traceability of sample activity
- Identification of activity recorded on the documents
- Error correction methods

9.3 Discussion of the On-Site Team's Findings

The EPA shall discuss the findings of the quality assurance and evidentiary auditors prior to debriefing the Contractor. During the debriefing, the auditors present their findings and recommendations for corrective actions necessary to the Contractor personnel.

9.4 Corrective Action Reports For Follow-Through to Quality Assurance and Evidentiary Audit Reports

Following an on-site laboratory evaluation, quality assurance and/or evidentiary audit, reports which discuss deficiencies found during the on-site evaluation may be sent to the Contractor. In a detailed letter, the Contractor shall discuss the corrective actions implemented to resolve the deficiencies discussed during the on-site evaluation and discussed in the report(s) to the EPA within 14 days of receipt of the report. An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of the Agency to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the EPA why he/she is unable to meet the delivery schedule listed in this section. The EPA will not grant an extension for greater than 14 days for the Contractor's response letter to the quality assurance and evidentiary audit report. The Contractor shall proceed and not assume that an extension will be granted until so notified by the EPA.

9.4.1 If new SOPs are required to be written, or if existing SOPs are required to be rewritten or amended because of the deficiencies and the subsequent corrective action implemented by the Contractor, the Contractor shall write/amend the SOPs per the requirements listed in Exhibit E, Section IV.

9.5 Corrective Actions

If the Contractor fails to adhere to the requirements listed in this section, the Contractor may expect, but the Agency is not limited to the following actions: reduction in the number of samples sent under the contract, suspension of sample shipment to the Contractor, an on-site laboratory evaluation, data package audit, a remedial performance evaluation sample, and/or contract sanctions, such as a Cure Notice.

10.0 DATA MANAGEMENT

Data management procedures are defined as procedures specifying the acquisition or entry, update, correction, deletion, storage and security of computer readable data and files. These procedures shall be in written form and contain a clear definition for all databases and files used to generate or resubmit deliverables. Key areas of concern include: system organization (including personnel and security), documentation operations, traceability and quality control.

10.1 Data manually entered from hard-copy shall be quality controlled and the error rates estimated. Systems should prevent entry of incorrect or out-of-range data and alert data entry personnel of errors. In addition, data entry error rates shall be estimated and recorded on a monthly basis by reentering a statistical sample of the data entered and calculating discrepancy rates by data element.

10.2 The record of changes in the form of corrections and updates to data originally generated, submitted, and/or resubmitted shall be documented to allow traceablilty of updates. Documentation shall include the following for each change:

- Justification or rationale for the change.
- Initials of the person making the change or changes. Data changes shall be implemented and reviewed by a person or group independent of the source generating the deliverable.
- Change documentation shall be retained according to the schedule of the original deliverable.
- Deliverables shall be reinspected as a part of the laboratory's internal inspection process prior to resubmission. The entire deliverable, not just the changes, shall be inspected.
- The Laboratory Manager shall approve changes to originally submitted deliverables.
- Documentation of data changes may be requested by laboratory auditors.

10.3 Lifecycle management procedures shall be applied to computer software systems developed by the laboratory to be used to generate and edit contract deliverables. Such systems shall be thoroughly tested and documented prior to utilization.

- A software test and acceptance plan including test requirements, test results and acceptance criteria shall be developed, followed, and available in written form.
- System changes shall not be made directly to production systems generating deliverables. Changes shall be made first to a development system and tested prior to implementation.
- Each version of the production system will be given an identification number, date of installation, and date of last operation and will be archived.
- System and operations documentation shall be developed and maintained for each system. Documentation shall include a user's manual and an operations and maintenance manual.

10.4 Individual(s) responsible for the following functions shall be identified:

- System operation and maintenance including documentation and training.
- Database integrity, including data entry, data updating and quality control.
- Data and system security, backup and archiving.